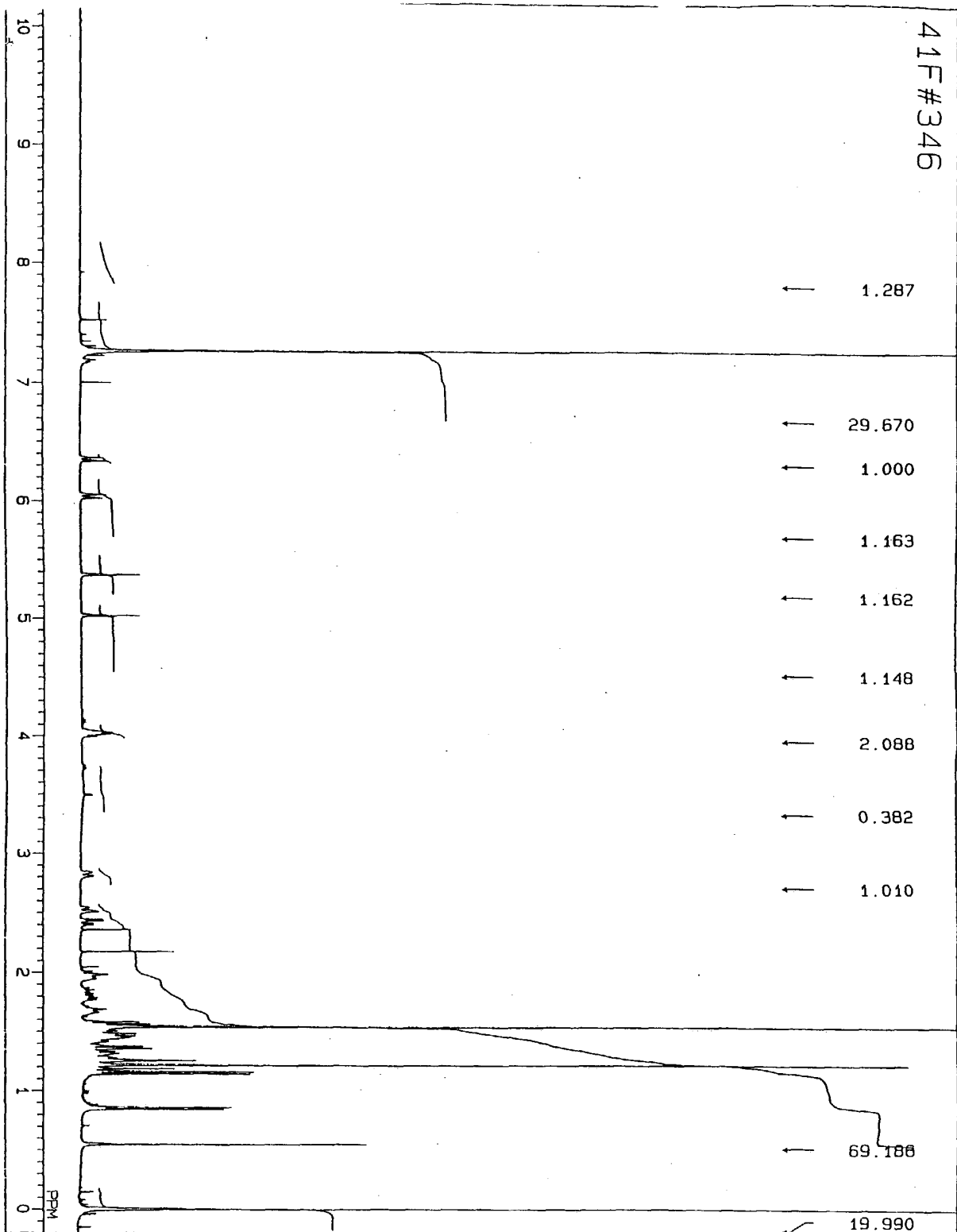


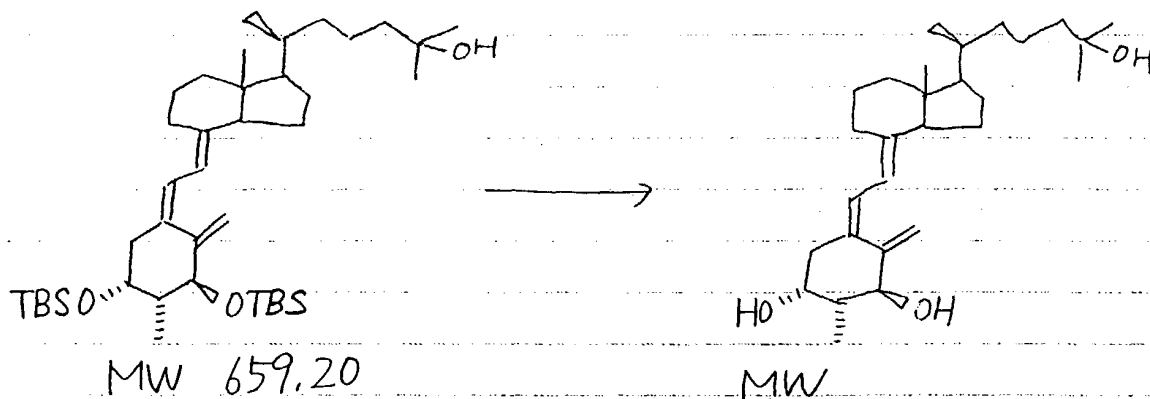
41F#346



SLVNT COCL3
 OBNUC 1H
 OBFREQ 399.65 MHz
 OBSET 124.00 KHz
 OBFIN 10905.1 Hz
 PWT 5.9 us
 POINT 32768
 SAMPD 32768
 SCANS 9216
 DUMMY 0
 FREQU 5000.0 Hz
 FILTR 5000 Hz
 ACQTM 3.277 sec
 PD 5.000 sec
 RGAIN 25
 BF 0.10 Hz
 T1 0.0 %
 T2 0.0 %
 T3 90.0 %
 T4 100.0 %
 EXMOD SGNON
 DFILE (100.140) FNO346
 SHMFL THS
 SPEED 15 Hz
 OPERATOR J.SHIWODE

4.5

#346



#345のwork up

CSA

MeOH

11mg

1ml

20:30~

1) 反応液から MeOH と

はし. 水を加え EA 抽出.

11:00

2) brine 洗い. MgSO₄ 上 脱水

3) 蒸気イボ.

シリカゲルカラムで分離後 ~~4.5mg~~

HPLC カラム (ODS #18) で分離.

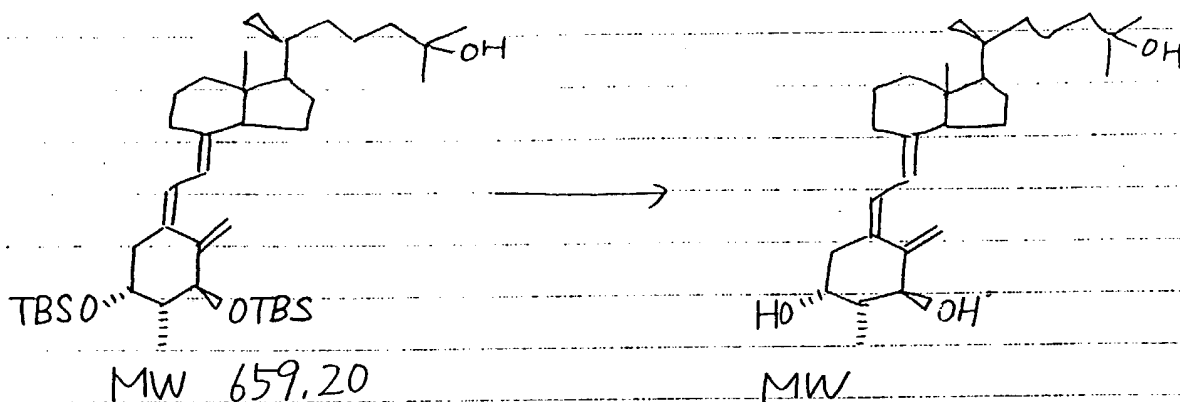
RP-18

4.5mg

(4.31%)

4.5

#346



{ #345のworkup
 CSA
 MeOH

11mg
 1ml

20:30~

{

 rtかくはん後 反応液から MeOH を

 とはし 水を加え EA 抽出

 brine 洗い MgSO₄ 上 脱水

 ろか、エバポ

 }

11:00

{

 シリカゲルカラムで分離後 ~~エバポ~~ 4.5mg

 HPLC カラム (ODS (18)) で分離 (y. 31%)

 RP-18

 }

{

 After stirring at rt, MeOH was evaporated from reaction mixture,

 water was added and extracted with EA

 washed with brine, dried over MgSO₄

 filtered, evaporated

 }

{

 After separation by silica gel column chromatography

 separation by HPLC column (ODS (18))

 }

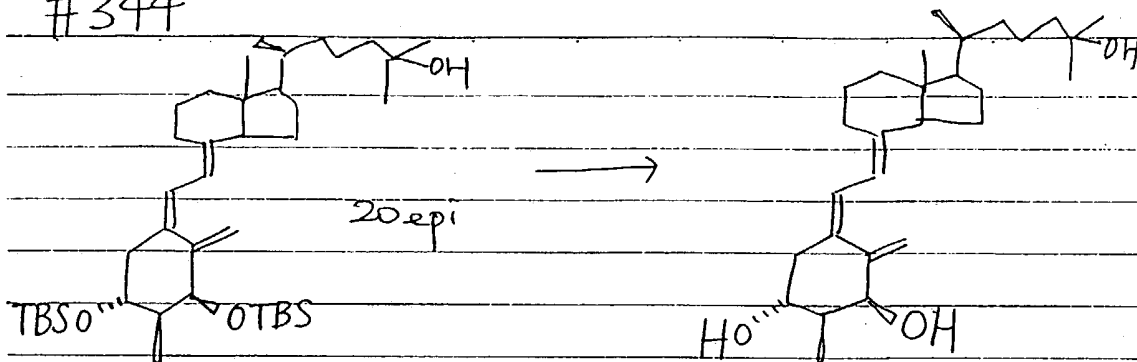
33.8904
9206

33.5847
594

30.2

10 mg

#344



MW

MW 430.67

#343 本體 work up

CSA MW 232.30 11 mg
MeOH 1 ml

ArE にかかると 14:20 ~
~50 ml

9:00

MeOHを留去し、水を加え、EA抽出、brine洗い。

MgSO₄上脱水。ろか！エバポレート。

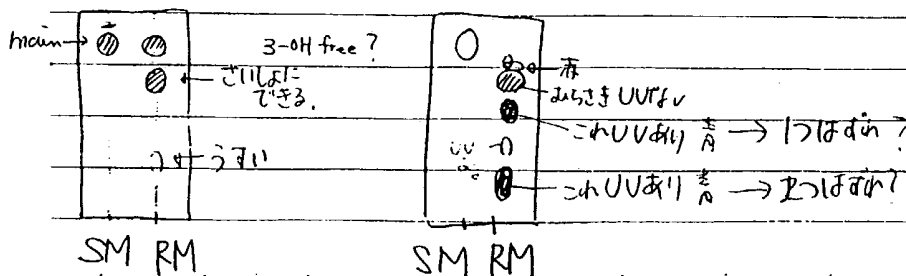
シリカゲルカラム (φ0.9 cm 10 cm height, EA=n-hex=1:1)

にて精製

9.3 mg (y. 63%)

→ HPLCにて分離。

EA=n-hex=1:1



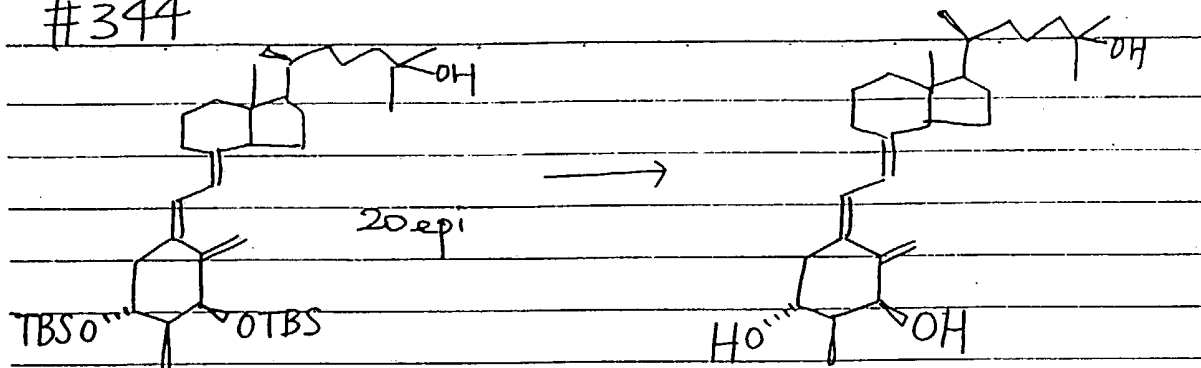
33.8904
9205

33.5847
594

60 mg

No.
cur

#344



MW

MW 430.67

[protective material]

{ #343 [木]体 work up

CSA MW 232.30

11 mg

[stirring under Ar at rt]

MeOH 1 ml

[Ar下 16:00 ~ 14:20 ~

9:00

~50 ml

MeOHを留去し、水を加え、EA抽出、brine洗う。

MgSO₄上脱水、ろ過、エバポレート。

シリカゲルカラム (φ0.9 cm 10 cm height, EA=n-hex = 1:1)

にて精製

9.3 mg (y. 63%)

→ HPLCにて分離

MeOH was distilled away, water was added, extracted with EA, washed with
dried over MgSO₄, dehydration, filtration, evaporation
purification by silica-gel column chromatography

EA=n-hex=1:1

(φ0.9 cm 10 cm height, EA/n-Hex = 1:1)

→ Separation by H

